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Speciation of Dissolved Selenium in the Kaoping and Erhjen Rivers and Estuaries, Southwestern Taiwan

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ABSTRACT: Analytical methods of dissolved Se species in river water and seawater were established and applied to study dissolved Se speciation in the Kaoping and Erhjen rivers and estuaries, southwestern Taiwan. The Kaoping and Erhjen rivers and estuaries were respectively in relatively oxygenated and oxygen-deficient conditions as revealed from the distributions of dissolved oxygen, DOC, nutrients, and dissolved Mn. Concentrations of dissolved total Se increased downstream in the riverine sections, ranging from 0.6 nM to 1.2 nM for the Kaoping River and from 0.8 nM to 1.05 nM for the Erhjen River. The dissolved total Se was only slightly higher in the Erhjen middle estuary than in the Kaoping middle estuary in spite of heavier pollution in the former. The dissolved total Se behaved rather conservatively in the Kaoping estuary but nonconservatively in the Erhjen estuary resulting from anthropogenic inputs, and in this respect showed similarities with the behavior of redox-sensitive Mn. The predominant species of selenium were Se(VI) and organic Se in the Kaoping River and Se(VI) in the Kaoping estuary. The elevated concentration of Se(VI) in the Kaoping estuary may stem from the degradation of organic Se and oxidation of Se(IV). On the other hand, Se(IV) and organic Se were equally dominant in the Erhjen River, and Se(IV) was predominant through most of the Erhjen estuary. The elevated distribution of Se(IV) in the Erhjen middle estuary may result mostly from partial decomposition of organic Se, but further oxidation of Se(IV) was inhibited in reducing waters. However, Se(VI) became dominant at Erhjen outer estuary where water was oxygenated by the replacement of intruded coastal seawater. Apparently, the speciation of dissolved Se was mainly controlled by the biological and redox processes in the Kaoping and Erhjen rivers and estuaries.

Introduction

Selenium is a metalloid element that usually exists in multiple oxidation states in natural waters. Three dissolved Se species, selenate(VI), selenite(IV), and organo-Se have been widely recognized in seawater (Robberecht and Van Grieken 1982; Cutter and Bruland 1984; Takayanagi and Wong 1985; Cutter 1989). The speciation of Se is of interest to environmentalists because certain species may be more available or toxic than others (Cutter 1982). Selenite, for instance, was found to enhance growth of a marine diatom in the laboratory (Price et al. 1987). Wrench and Measures (1982) reported that selenite was preferentially taken up by phytoplankton during periods of high primary productivity in coastal seawater. Apte and Howard (1986) also found depletion of Se(IV) in a closed experimental ecosystem during the development of a phytoplankton bloom. The major source of selenium in coastal seawater is generally

believed to come from riverine fluxes (Cutter 1991).

Biogeochemical processes occurring during the mixing of river water and seawater may determine the speciation and behavior of dissolved elements in estuaries (Liss 1976; Sholkovitz and Copland 1981; Takayanagi and Wong 1984; Church 1986). The presence of selenium species therefore may be critical in determining the availability and transport of Se through rivers and estuaries as well as the fate of Se in the coastal ecosystems. The speciation and behavior of Se appear to show considerable variation probably due to the contrasting biogeochemical processes of Se in various estuaries. For example, Measures and Burton (1978) reported that Se behaved conservatively in the Test River at salinities greater than 7‰ and that Se(IV) was the minor species. In contrast, Takayanagi and Wong (1984) and Takayanagi and Cossa (1985) reported that Se(IV) was the major species and removed from the James and upper St. Lawrence estuarine waters, respectively. Cutter (1989) found that dissolved Se was conservative in the South San Francisco Bay but nonconservative in the North

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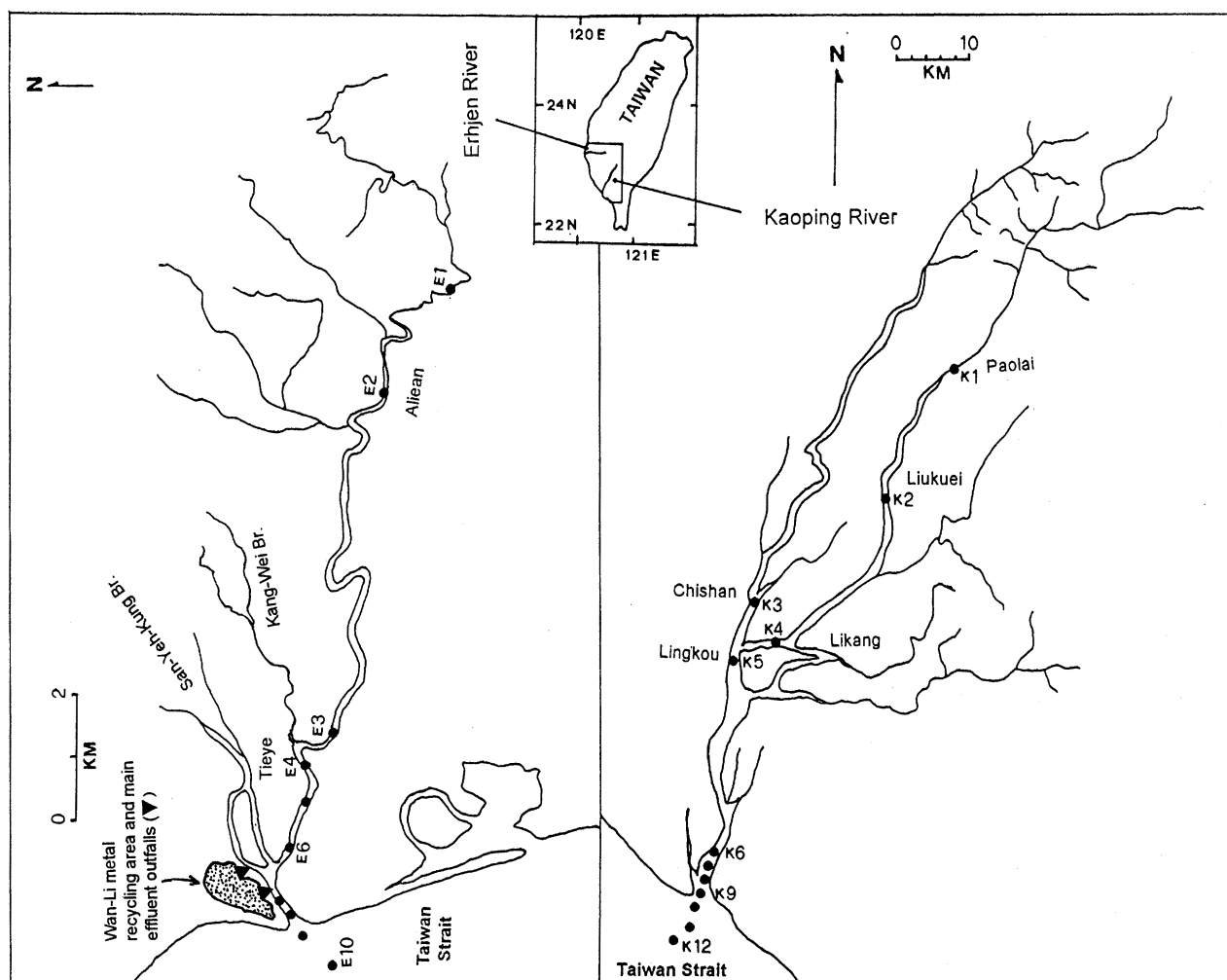


Fig. 1. Sampling locations in the Kaoping and Erhjen rivers and estuaries.

San Francisco Bay, and in general, Se(VI) was the predominant species. Apparently, further studies are necessary in order to understand the distribution of Se species in Taiwan's rivers and estuaries, and to understand the mechanisms controlling the speciation of Se in riverine and estuarine waters in general.

Materials and Methods

STUDY AREA

The Kaoping and Erhjen rivers are located in southwestern Taiwan (Fig. 1). The Kaoping River is the second largest in Taiwan and has been increasingly polluted by industrial and domestic wastewater in recent years (Hung and Huang 1990). The drainage basin and annual discharge of the river are 3,257 km² and 1.23×10^{10} m³ yr⁻¹, respectively (Water Resources Planning Commission 1990). The Erhjen River is relatively small but

seriously contaminated with copper, zinc, nitric acid and organic pollutants from domestic effluents (discharged from San Yeh-Kang and Kang-Wei branches) and industrial effluents (discharged from Wan-Li metal recycling county) in the upper to middle estuarine regions (Wei 1983; Hung et al. 1987; Hung and Tsai 1992). The drainage basin and annual discharge of the river are 350 km² and 3.53×10^8 m³ yr⁻¹, respectively (Water Resources Planning Commission 1990).

SAMPLING AND ANALYTICAL METHODS

Water samples (river and estuarine) were collected at 25 cm below the surface with a pre-cleaned horizontal water sampler (Ekman Birge) during the dry season in October 1990 for the Kaoping River and November 1990 for the Erhjen River. The riverine samples were collected from highway bridges. Estuarine samples were collected

aboard the fishing vessel in an upstream direction from a frontal position on the vessel to minimize the risk of contamination. Four liters of each sample were taken (without leaving any air space) in acid-cleaned PE bottles at 4°C and immediately transported back to the laboratory for further pre-treatment. In addition, pH, salinity, and dissolved oxygen were measured in situ with a portable pH meter (Orion Research), DO meter (YSI model 58), and salinometer (Hydro-Bios). In the laboratory, the water samples were filtered through acid-cleaned filters (Nucleopore, 0.4 μm) in a nitrogen-filled plastic bag for determinations of Se, Mn, and nutrients. Another 500 ml was filtered through a precombusted (450°C, 4 h) GF/F filter, and a final portion of 50 ml was collected for DOC measurement. DOC was measured with a TOC analyzer (O. I. Corporation) with improved procedures for seawater (Hung and Chang 1992). Nutrients were determined using the methods of Strickland and Parsons (1972).

The determination of Se species was carried out in a clean room (class 1,000) equipped with a chemical hood of class 100. The analytical procedures (schematically illustrated in Fig. 2) were as follows: The organic Se species were preconcentrated by passing a 500-ml subsample, with the pH successively adjusted to 8.0 and 3.0, through separated column sets, where each set comprised two connected C_{18} Sep-Pak cartridges (Waters Association, Milford, USA). The species preconcentrated at pH 8.0 and 3.0 were classified as neutral/basic and acidic organic Se, respectively (Sugimura et al. 1978). The column sets were then eluted with purified methanol (10 ml) and the eluents were combined together in a Teflon cup before drying on a hot plate. The residue was dissolved in diluted ultrapure HNO_3 (2%, v/v) solution for determination of organic Se. The water sample passing through the columns was acidified to pH 1.2 with ultrapure HCl , after which the selenite in the solution was preconcentrated by complexation with 2 ml of ammonium pyrrolidinedithiocarbamate (5%, v/v) followed by adsorption of complexes on the C_{18} column (Sturgeon et al. 1985). The extracted Se(IV) was eluted and purified according to the procedures described above for organic Se. Finally, the organic Se-extracted and selenite-extracted solution was measured for selenate using the procedures developed by Takayanagi and Wong (1985): adding 3 ml of Tellurium carrier solution (2%, v/v), 2 g of hydrazine sulfate, and 150 ml of 12 M HCl in a 250 ml solution, which was then gently boiled for 15 min, cooled, and filtered through a 0.22- μm filter (Nucleopore). The residue on the filter was dissolved in 5 ml of 2% HNO_3 for Se(VI) determination. The concentration of each species

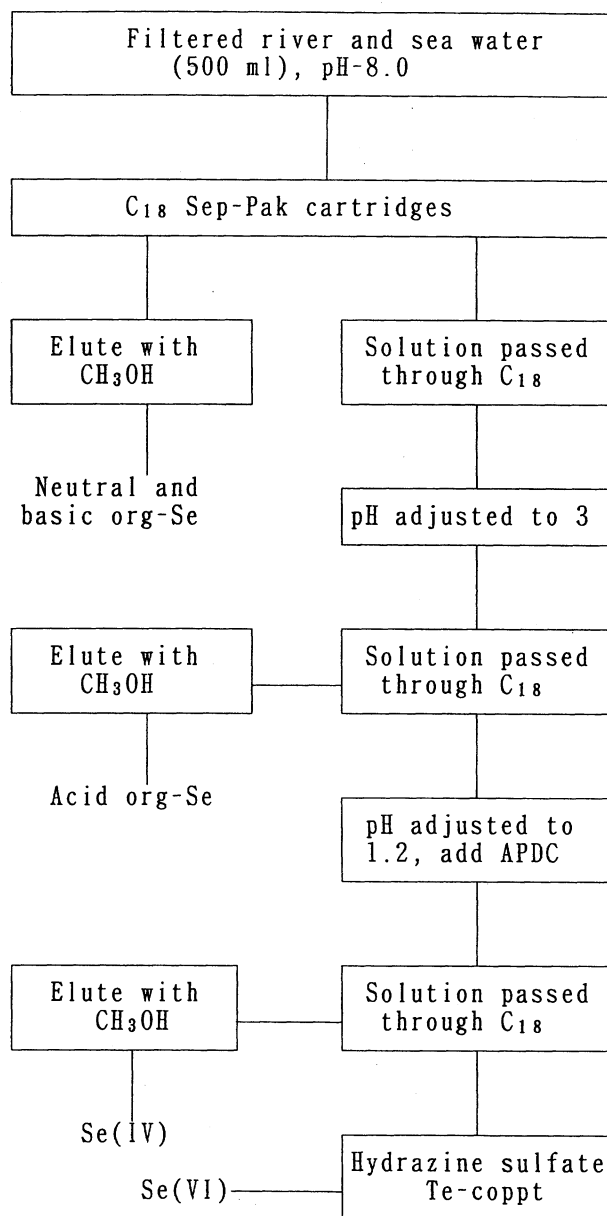


Fig. 2. Schematic procedures for the analyses of organic Se, selenite, and selenate.

in natural samples is derived from the mean of triplicate analyses.

Dissolved Mn was preconcentrated by complexing Mn with 8-hydroxyquinoline at pH 8.5–9.0 followed by adsorption of complexes on C_{18} Sep-Pak cartridges. The preconcentrated Mn was then eluted with methanol, dried, and dissolved in dilute HNO_3 (2%) solution. Each sample was also run in triplicate.

The preconcentrated Se species and Mn were determined with a Perkin-Elmer 5100 PC atomic

TABLE 1. Addition and recovery of Se (IV), Se (VI), and Mn from natural river and sea waters.

Species	Matrix	Spike ($\mu\text{g l}^{-1}$)	Preconcentration Method	Replicate	Recovery %	Standard Deviation %
Se (IV)	River water	5.0	APDC/C ₁₈	6	96.9	1.1
Se (IV)	River water	5.0	Coprecipitation	6	97.5	2.1
Se (VI)	River water	5.0	Coprecipitation	6	94.9	1.5
Mn (II)	River water	10	Oxine/C ₁₈	6	93.1	1.7
Se (IV)	Seawater	5.0	APDC/C ₁₈	6	95.1	1.2
Se (IV)	Seawater	5.0	Coprecipitation	6	98.2	0.8
Se (VI)	Seawater	5.0	Coprecipitation	6	96.2	1.9
Mn (II)	Seawater	10	Oxine/C ₁₈	6	94.2	1.4

absorption spectrophotometer equipped with an HGA 600 graphite furnace. Matrix modifier, Zeeman background correction, and measuring conditions slightly modified from those recommended by the manufacturer were applied throughout the measurements. Recoveries of Se(IV), Se(VI), and Mn were evaluated by spiking in natural riverwater and seawater and were generally better than 5%, 5%, and 7%, respectively, as shown in Table 1. Duplicate analyses of Se(IV) for a natural seawater reference (NASS-2, National Research Council Canada) were 11% deviation from the certified value (0.34 nM). Organic Se determined in this study was verified from the difference between total Se (determined with the method of Measures and Burton 1980) and inorganic Se(IV + VI) for a Kaoping estuarine water (salinity = 5.2‰). The difference was estimated to be 9% ($n = 4$).

Results and Discussion

Distributions of pH, dissolved oxygen, and dissolved organic carbon in the Kaoping and Erhjen rivers and estuaries are shown in Fig. 3 and Fig. 4, respectively. Dissolved oxygen was generally greater than 186 μM in Kaoping riverine and estuarine waters, with the exception of a relatively low concentration at the K5 station (141 μM) that apparently resulted from the influence of human activity. The

distribution of pH varied only slightly from 8.0 through the Kaoping River and estuary. DOC was generally inversely related to dissolved oxygen, probably due to a causal relationship between high organic matter concentrations and high biochemical oxygen demand. DOC showed a slight removal in the Kaoping estuary, possibly related to its microbial degradation. The features in the Erhjen system were quite different. Due to discharge of acid and organic wastes into the upper to middle estuary from Wen-Li metal recycling area and two highly polluted branches (San Yeh-Kung and Kang-Wei), pH and dissolved oxygen decreased dramatically in the middle estuary where DOC displayed a maximum value (Fig. 4). The different character of the two river-estuary systems is also reflected in the distribution of nutrients (Figs. 5 and 6). Phosphate and silicate indicated more or less conservative mixing in both estuaries, whereas nitrate plus nitrite was conservative in the Kaoping estuary but removed in the upper, or added in the middle, ranges of the Erhjen estuary due to biomediated loss and anthropogenic inputs, respectively.

The distribution of dissolved Mn can be also used as an indicator of the redox condition of riverine and estuarine waters. The concentration of dissolved Mn was more than one order magnitude higher in the Erhjen river-estuary (Fig. 7) than in

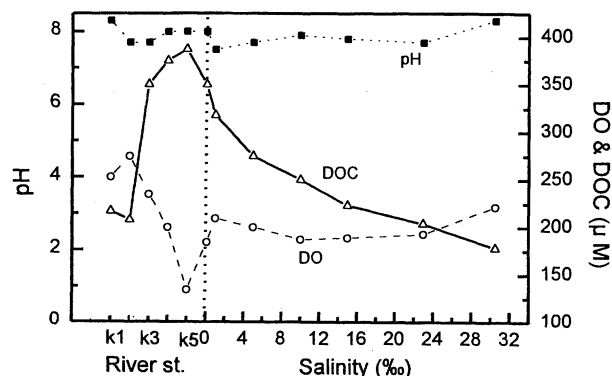


Fig. 3. Distributions of pH, dissolved oxygen, and dissolved organic carbon in the Kaoping River and estuary.

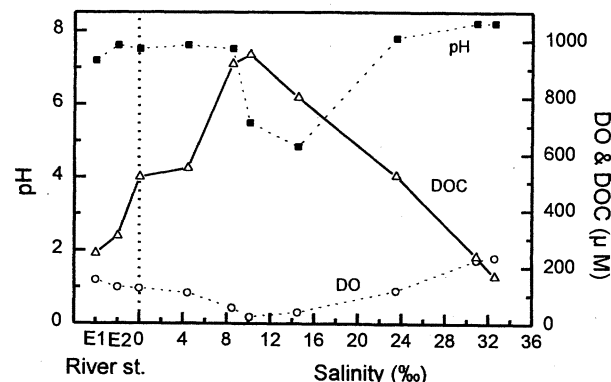


Fig. 4. Distributions of pH, dissolved oxygen, and dissolved organic carbon in the Erhjen River and estuary.

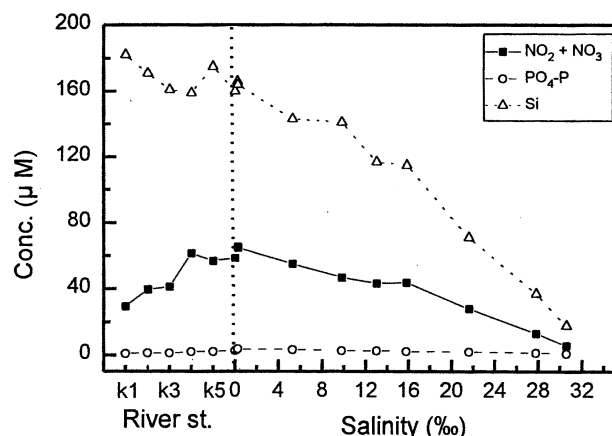


Fig. 5. Distributions of dissolved nitrogen (nitrite + nitrate), phosphate, and silicate in the Kaoping River and estuary.

the Kaoping river-estuary (Fig. 8). This was probably due to the enhanced reductive dissolution of naturally occurring Mn oxides in the more polluted Erhjen system but may in part also have reflected direct anthropogenic Mn inputs. Whatever the case, the dissolved Mn data support the DO distributions in suggesting that conditions were significantly more reducing in the Erhjen system.

The distributions of total selenium (Σ of individually determined Se species) in the Kaoping River and estuary are shown in Fig. 8. The concentration of total dissolved Se in the Kaoping River increased downstream, ranging from 0.6 nM at the upper stream site to 1.2 nM at the river-estuary interface, which apparently resulted from the influence of river basin runoff and human inputs. The concentration then decreased down the estuary to 0.48 nM at 30.5‰. Although a seawater end-member was not sampled, in another study we did find about 0.38 nM of total Se in the coastal seawater

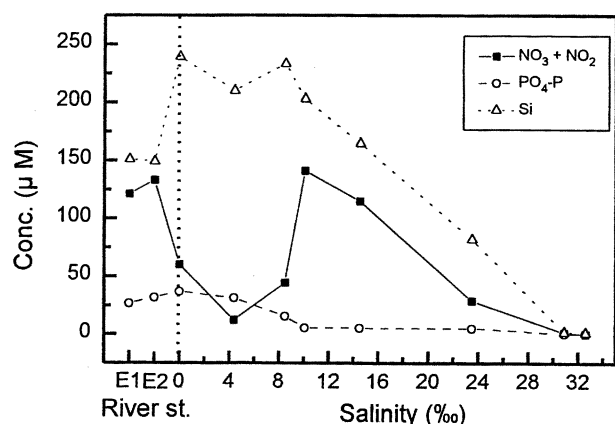


Fig. 6. Distributions of dissolved nitrogen (nitrite + nitrate), phosphate, and silicate in the Erhjen River and estuary.

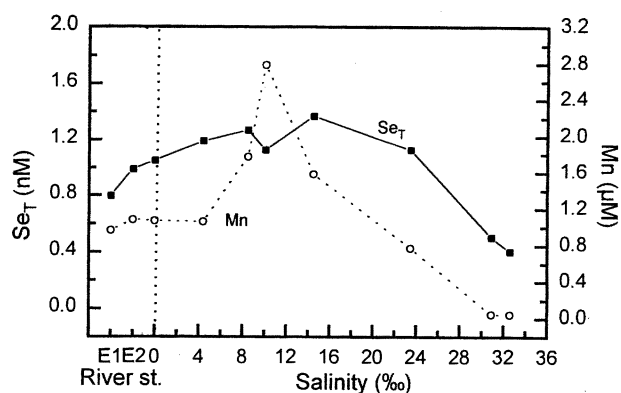


Fig. 7. Distributions of dissolved Mn and total dissolved Se in the Erhjen River and estuary.

(salinity = 34.2‰) of southeastern Taiwan Strait. Apparently, the distribution of total Se in the Kaoping estuary was conservative. The correlation coefficient (0.99) between salinity and total Se was highly significant with a slope of $-0.027 \text{ nM ml}^{-1}$.

As for the Se speciation, organic Se (34–52% of total) and Se(VI) (22–37% of total) were dominant over the Se(IV) (20–28% of total) in the lower reaches of the Kaoping River (Fig. 9), probably related to the high DOC but well oxygenated condition. In the Kaoping estuary, organic Se decreased from 0.63 nM (53% of total) at river end-member to 0.06 nM (14% of total) at 30.5‰. The decrease of organic Se in the estuary was closely related to the decrease of DOC. The microbial degradation of DOC may be responsible for the decrease of organic Se, and the decomposed organic Se may be subsequently oxidized to Se(VI). The distribution of Se(IV), which amounted to 8–15% of total Se, was also nonconservative in the estuary where Se(IV) may be removed through biological uptake or it may be oxidized to Se(VI) under oxygenated conditions. The utilization of

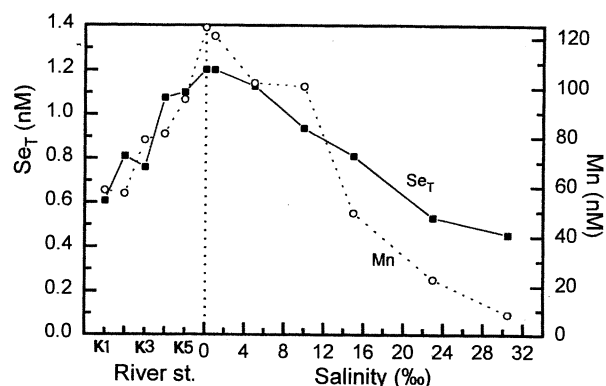


Fig. 8. Distributions of dissolved Mn and total dissolved Se in the Kaoping River and estuary.

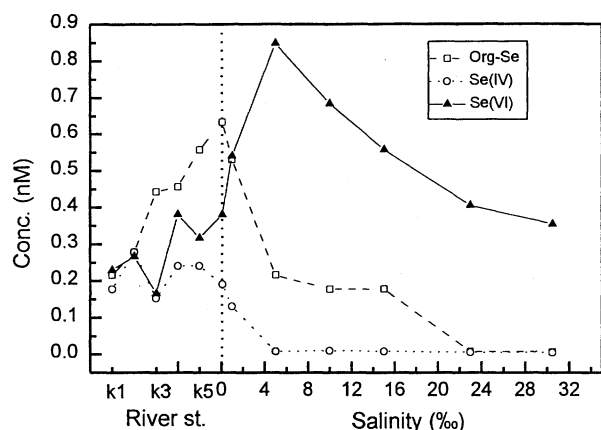


Fig. 9. Selenium speciation in the Kaoping River and estuary.

Se(IV) by marine phytoplankton has been confirmed by Wrench (1978), Wrench and Measures (1982), Apte and Howard (1986), and Price et al. (1987); whereas the oxidation of Se(IV) was reported by Takayanagi and Wong (1984). Masscheleyn et al. (1990) also proposed that Se(IV) can be slowly oxidized to Se(VI) if Eh is above 200 mV. Because Se(IV) and Se(VI) were analyzed independently, the contrasting distributions between the two species supports the hypothesis that the nonconservative addition of Se(VI) with respect to salinity in the estuary was most likely derived from the oxidation of organic Se and Se(IV). The fraction of the predominant species of Se(VI) in the estuarine ranges was approximately from 32% to 78%.

In order to support the above findings, an estuarine model (Kual and Froelich 1984) was applied to evaluate the mass-balance among the Se species in the Kaoping estuary, which can be considered as a simple system because Kaoping river water is the major Se source. Linear relationships between concentrations of Se species and salinities were derived using the data of two end-members (river water and seawater). Estuarine distributions of Se species deviated positively or negatively from this linearity. The deviations were integrated over the estuarine section to show the magnitude of addition or removal in the estuary as a whole. The magnitude of removal (Se(IV) + organic Se) was found to be 27.2% greater than that of addition (Se(VI)). However, particulate Se was found to compose up to 24.7% with respect to the inventory, derived from conservative mixing in the estuary, using the data of Shy (1991). Assuming the particulate Se was derived from uptake of Se(IV) by biological organisms as generally thought previously (Wrench and Measures 1982; Apte and Howard 1986; Price et al. 1987), the mass-balance

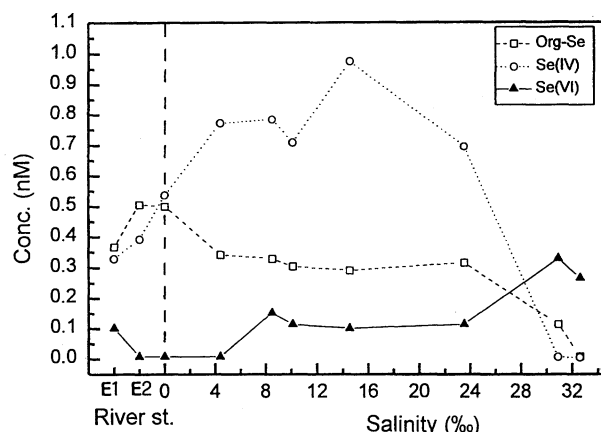


Fig. 10. Selenium speciation in the Erhjen River and estuary.

argument therefore supports the view that the elevated Se(VI) in the estuary was derived from the oxidation of Se(IV) and organic Se.

The distribution of total Se (Σ of individually determined Se species) in the Erhjen River is shown in Fig. 7. The concentration increased downstream in the river, ranging from 0.8 nM to 1.05 nM, which is comparable to the situation in the Kaoping River. However, the nonconservative distribution (addition) of total Se in the estuary as was aforementioned. The Se speciation (Fig. 10) shows that Se(IV) and organic Se were equally distributed (41–51% of total) in the Erhjen River but Se(IV) was clearly dominant (62–71% of total) over organic Se and Se(VI) in the most regions of the estuary. The elevated distribution of Se(IV) in the middle estuary may result mostly from anthropogenic inputs and the decomposition of organic Se, but further oxidation of Se(IV) was inhibited in reducing estuarine waters. It was also possible that further reduction of Se(IV) to elemental Se(0) in particulate form took place. Meanwhile, Erhjen estuary received relatively small river discharge and high tidal prism compared with the Kaoping estuarine conditions during the estuarine samplings, which may result in the dilution effect of Se in the Erhjen estuary. These situations may partially explain why the concentrations of total dissolved Se were similar in the two estuaries, in spite of the fact that the contamination was more serious in the Erhjen estuary than in the Kaoping estuary. Distributions of organic Se in the Erhjen estuary did not show significant removal and concentrations were generally higher than those in the Kaoping estuary. This may be due to the fact that the degradation of organic Se was relatively inefficient under less well oxygenated conditions. The concentration of Se(VI) was lower than those of

Se(IV) and organic Se in the Erhjen River and estuary except that Se(VI) was dominant (ca 65% of total) in the outer estuary, where the water became oxygenated and Se(IV) was oxidized to Se(VI) again.

The use of a simple estuarine model to elucidate the speciation of Se in the Erhjen estuary is not feasible because Se inputs other than riverine source cannot be quantified. However, total Se determined (in duplicate) directly with the method of Measures and Burton (1980) was used to compare with the sum of Se species. The significant correlation ($R = 0.981$, $p < 0.01$) and nearly identical magnitudes (slope = 0.932) strongly suggest that the summed Se species is equivalent to the total Se, which may primarily result from geochemical and anthropogenic processes. However, the concentration of a Se species can be largely determined by biological and redox processes, which apparently govern the interconversion of Se species.

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